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(54) Element with antistat layer

(57) An imaging element comprising: a support; at least one image forming layer; and an antistat layer, wherein said antistat layer comprises: a chlorinated polyolefin and a conductive agent.

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[0008] Although the prior art is replete with patents disclosing various antistatic backings for photographic paper (for example, US Patent Nos. 3,671,248; 4,547,445; 5,045,394; 5.156,707; 5,221,555; 5,232,824; 5,244,728; 5,318,886; 5,360,707; 5,405,907 and 5,466,536), not all of the aforesaid issues are fully addressed by these inventions. Also, some of the inventions of the prior art may alleviate one or more problems but may aggravate some others. For example, US Patent No. 3,525,621 teaches that antistatic properties can be given to an aqueous coating composition by practically any silica sol, but preferably a silica of large surface area of the order of 200-235 m^2/g in combination with an alkylaryl polyether sulfonate. However, the high solubility of the alkylaryl polyether sulfonate in aqueous medium causes leaching during processing resulting in poor backmark retention of such antistatic layers. Similarly, US Patent No. 5,244,728 teaches a binder polymer consisting of an addition product of alkyl methacrylate, alkali metal salt and vinyl benzene which, when incorporated in an antistatic layer for photographic paper, substantially improves backmark retention characteristics but compromises spliceability and track-off characteristics, as demonstrated in US Patent No. 5,683,862. US Patent No. 5,466,536 teaches the use of a mixture of polymers and copolymers with specific acrylic acid content for good printabilty. However, the high acid number of these polymers make the antistatic layer (or debris thereof) vulnerable for softening in high pH developer solution, and can cause formation of soft tar-like species discussed herein above.

[0009] Moreover, backings developed for one type of polyolefin-coated paper may fail on a different type of polyolefin-coated paper. Therefore, although claims are generally made for both polyethylene and polypropylene coated photographic paper, a vast majority of patents in the art provide examples involving polyethylene coated photographic paper only, and the successful application of these teachings on polypropylene coated photographic paper is often, and even generally, not possible. In general, good adhesion of antistatic layers on a polypropylene surface is more difficult to achieve than on a polyethylene surface. For example, in US Patent No. 4,547,445 a layer containing gelatin and an inorganic pigment is claimed to have ink-retaining characteristics with good adhesion to polyethylene-coated photographic paper. But, as discussed in US Patent No. 5,853,965, such a gelatin containing layer is expected to fail adhesion on a biaxially oriented polypropylene-coated photographic paper. In fact, adhesion of auxiliary layers to polypropylene surfaces has become a key issue for reflective print media, as more and more products comprising such a surface are being disclosed in the patent literature and introduced to the market (vide, for example, US Patent Nos. 5,853,965; 5,866,282; and 5,874,205). Antistatic layers containing a styrene-maleic anhydride copolymer, colloidal silica and crosslinking compounds containing ethyleneimino groups and/or epoxy rings are disclosed in US Patent No. 4,266,016, allegedly for good antistatic characteristics and adhesion on both polyethylene and polypropylene surfaces. However, as demonstrated through comparative samples in US Patent No. 6,171,769, such antistatic layers provide neither the backmark retention characteristics nor the spliceability currently desired of photographic paper. US Patent No. 6,171,769, by itself, teaches of binder polymers with excellent adhesion to polypropylene surfaces. However, these binder polymers are not known to have any appreciable electrical conductivity, and, thus do not participate in antistatic function by themselves. Typically, for a given dry coverage of the antistatic layer, the higher the amount of binder polymer the better is the adhesion but poorer is the electrical conductivity of the layer.

[0010] A vast majority of antistatic formulations designed for use in photographic reflective media are aqueous based coating compositions, utilizing salts for ionic conductivity, inorganic particles such as colloidal silica as fillers and latex polymers as binders. Although the salt is needed for electrical conductivity, its presence can adversely affect the dispersion of the latex and/or the colloidal filler, through charge screening. Such an adverse effect unacceptably increases the viscosity of the coating composition and/or its shelf life, rendering it impractical for robust manufacturing. A careful balance needs to be struck in the content of the various ingredients to maintain appropriate viscosity and yet achieve the physical properties, such as conductivity and adhesion to the substrate, required of the resultant antistatic layer. In this context, identification of a binder polymer, which adds to the electrical conductivity (and, therefore, requires less salt) as well as provides good adhesion to the support appears highly desirable.

[0011] Thus, it is clear that the prior art does not fully meet the high demands and the diverse needs of the industry and requires further innovation. What the art needs is an antistatic backing for photographic elements, particularly reflective print media, wherein the antistatic layer provides superior electrical conductivity, backmark retention, spliceability and dusting characteristics through improved adhesion to the support.

[0012] The present invention provides an imaging element that overcomes the problems discussed above. The present invention to provide an imaging element, particularly, one comprising a reflective support, with an improved antistatic layer with excellent adhesion to a polypropylene, particularly biaxially oriented polypropylene, surface. The antistatic layer shows minimal dusting and, when used as a backing layer, provides improved backmark retaining characteristics. Furthermore, the antistatic layer is spliceable in typical high speed photofinishing equipment.

[0013] These and other objects of the invention are achieved by providing:

an imaging element comprising:

a support;

without other polymeric binders. Such other polymeric binders can include one or more of a water soluble polymer, a hydrophilic colloid or a water insoluble polymer, latex or dispersion. Particular preference is given to polymers selected from the group of polymers and interpolymers prepared from ethylenically unsaturated monomers such as styrene, styrene derivatives, acrylic acid or methacrylic acid and their derivatives, olefins, (meth)acrylonitriles, itaconic acid and its derivatives, maleic acid and its derivatives, vinyl halides, vinylidene halides, and others. Also included are aqueous dispersions of condensation polymers such as polyurethanes and polyesters. Also useful are primary amine addition salt interpolymers, specifically, the interpolymers that contain a polymerized vinyl monomer having a primary amine addition salt component. The most preferred polymeric binders to be used in conjunction with the chlorinated polyolefin of the present invention are those disclosed in U.S. Pat. Nos. 6,171,769 and 6,077,656.

[0022] The weight % of the chlorinated polyolefin in the dried antistatic layer can vary according to specific need but is preferred to be at least 1% and more preferred to be at least 3% and most preferred to be at least 5% to achieve desirable properties.

[0023] In addition to the chlorinated polyolefin, the antistatic layer of the present invention can comprise other electrically conductive agent(s), which can include any of the electronic and ionic conductive agents known in the art.

[0024] As mentioned earlier, the conductivity of antistatic layers employing an electronic conductor depends on electronic mobility rather than ionic mobility and is independent of humidity. Electronic conductors such as conjugated conducting polymers, conducting carbon particles, crystalline semiconductor particles, amorphous semiconductive fibrils, and continuous conductive metal or semiconducting thin films can be used in this invention to afford humidity independent, process-surviving antistatic protection. Of the various types of electronic conductors, electronically conductive metal-containing particles, such as semiconducting metal oxides, and electronically conductive polymers, such as, substituted or unsubstituted polythiophenes, substituted or unsubstituted polypyrroles, and substituted or unsubstituted polyanilines are particularly effective for the present invention.

[0025] Electronically conductive particles, which may be used in the present invention include conductive crystalline inorganic oxides, conductive metal antimonates, and conductive inorganic non-oxides. Crystalline inorganic oxides may be chosen from zinc oxide, titania, tin oxide, alumina, indium oxide, silica, magnesia, barium oxide, molybdenum oxide, tungsten oxide, and vanadium oxide or composite oxides thereof, as described in, e.g., U.S. Pat. Nos. 4,275,103; 4,394,441; 4,416,963; 4,418,141; 4,431,764; 4,495,276; 4,571,361; 4,999,276 and 5,122,445. The conductive crystalline inorganic oxides may contain a "dopant" in the range from 0.01 to 30 mole percent, preferred dopants being aluminum or indium for zinc oxide; niobium or tantalum for titania; and antimony, niobium or halogens for tin oxide. Alternatively, the conductivity can be enhanced by formation of oxygen defects by methods well known in the art. The use of antimony-doped tin oxide at an antimony doping level of at least 8 atom percent and having an X-ray crystallite size less than 100 Å and an average equivalent spherical diameter less than 15 nm but no less than the X-ray crystallite size as taught in U.S. Pat. No. 5,484,694 is specifically contemplated. Particularly useful electronically conductive particles which may be used in the antistatic layer include acicular doped metal oxides, acicular metal oxide particles, acicular metal oxides containing oxygen deficiencies, acicular doped tin oxide particles, acicular antimony-doped tin oxide particles, acicular niobium-doped titanium dioxide particles, and the like. The aforesaid acicular conductive particles preferably have a cross-sectional diameter less than or equal to 0.02 μm and an aspect ratio greater than or equal to 5:1.Some of these acicular conductive particles, useful for the present invention, are described in U.S. Pat Nos. 5,719,016; 5,731,119; 5,939,243 and references therein.

[0026] If used, the volume fraction of the acicular electronically conductive particles in the dried antistatic layer of the invention can vary from 1 to 70% and preferably from 5 to 50% for optimum physical properties. For non-acicular conductive metal oxides, the volume fraction can vary from 15 to 90%, and preferably from 20 to 80% for optimum properties.

[0027] The invention is also applicable where the conductive agent comprises a conductive "amorphous" gel such as vanadium oxide gel comprised of vanadium oxide ribbons or fibers. Such vanadium oxide gels may be prepared by any variety of methods, including but not specifically limited to melt quenching as described in U.S. Pat. No. 4,203,769, ion exchange as described in DE 4,125,758, or hydrolysis of a vanadium oxoalkoxide as claimed in WO 93/24584. The vanadium oxide gel is preferably doped with silver to enhance conductivity. Other methods of preparing vanadium oxide gels which are well known in the literature include reaction of vanadium or vanadium pentoxide with hydrogen peroxide and hydrolysis of VO_2 OAc or vanadium oxychloride.

[0028] Conductive metal antimonates suitable for use in accordance with the invention include those as disclosed in, U.S. Pat. Nos. 5,368,995 and 5,457,013, for example. Preferred conductive metal antimonates have a rutile or rutile-related crystallographic structures and may be represented as M^{+2} $Sb^{+5}{}_2$ O_6 (where M^{+2} =Zn^{+2}, Ni^{+2}, Mg^{+2},Fe^{+2}, Cu^{+2}, Mn^{+2}, Co^{+2}) or M^{+3} Sb^{+5} O_4 (where M^{+3} =In^{+3}, Al^{+3}, Sc^{+3}, Cr^{+3}, Fe^{+3}).
Several colloidal conductive metal antimonate dispersions are commercially available from Nissan Chemical Company in the form of aqueous or organic dispersions. Alternatively, U.S. Pat. Nos. 4,169,104 and 4,110,247 teach a method for preparing M^{+2} $Sb^{+5}{}_2$ O_6 by treating an aqueous solution of potassium antimonate with an aqueous solution of an appropriate metal salt (e.g., chloride, nitrate, sulfate, etc.) to form a gelatinous precipitate of the corresponding insoluble

less than 70 nm and most preferably less than 40 nm. A variety of colloidal sols useful in the present invention are commercially available from DuPont, Nalco Chemical Co., and Nyacol Products Inc.

[0037] The weight % of the inorganic particles of the aforesaid sol are preferred to be at least 5% and more preferred to be at least 10% of the dried antistatic layer of the invention to achieve the desired physical properties.

[0038] Other optional addenda that may be incorporated in the antistatic layer of the present invention include tooth-providing ingredients (vide US Patent No. 5,405,907, for example), colorants, crosslinking agents, surfactants and coating aids, defoamers, thickeners, coalescing aids, matte beads, lubricants, pH adjusting agents, plasticizers, and other ingredients known in the art.

[0039] The dry coverage of the antistatic layer of the present invention can be from 10 mg/m^2 to 10,000 mg/m^2, but preferably from 100 mg/m^2 to 1000 mg/m^2.

[0040] The coating solution for forming the antistatic layer of the present invention can be aqueous, non-aqueous or mixtures thereof; however, aqueous solutions are preferred for environmental reasons. The surface on which the coating solution is deposited for forming the antistatic layer can be treated for improved adhesion by any of the means known in the art, such as acid etching, flame treatment, corona discharge treatment, glow discharge treatment, etc. or can be coated with a suitable primer layer. However, corona discharge treatment is the preferred means for adhesion promotion.

[0041] The antistatic layer of the invention can be formed on any polymer sheet, with particular preference for those, which are known for their application as supports in imaging elements. The polymer sheet can comprise homopolymer(s), copolymer(s) or interpolymer(s) and/or mixtures thereof. Typical imaging supports comprise cellulose nitrate, cellulose acetate, poly(vinyl acetate), polystyrene, polyolefins including polyolefin ionomers, polyesters including polyester ionomers, polycarbonate, polyamide, polyimide, glass, natural and synthetic paper, resin-coated or laminated paper, voided polymers including polymeric foam, microvoided polymers and microporous materials, or fabric, or any combinations thereof. Preferred polymers are polyesters, polyolefins and polystyrenes, mainly chosen for their desirable physical properties and cost.

[0042] Suitable polyolefins include polyethylene, polypropylene, polymethylpentene, polystyrene, polybutylene and mixtures thereof. Polyolefin copolymers, including copolymers of propylene and ethylene such as hexene, butene and octene and mixtures thereof are also useful.

[0043] The polymer sheet can comprise a single layer or multiple layers according to need. The multiplicity of layers may include any number of auxiliary layers such as other antistatic layers and backmark retention layers, tie layers or adhesion promoting layers, abrasion resistant layers, curl control layers, cuttable layers, conveyance layers, barrier layers, other splice providing layers, UV absorption layers, antihalation layers, optical effect providing layers, waterproofing layers, flavor retaining layers, fragrance providing layers, adhesive layers, imaging layers and the like.

[0044] The polymer sheet can be formed by any method known in the art such as those involving extrusion, coextrusion, quenching, orientation, heat setting, lamination, coating and solvent casting. It is preferred that the polymer sheet is an oriented sheet formed by any suitable method known in the art, such as by a flat sheet process or a bubble or tubular process. The flat sheet process involves extruding or coextruding the materials of the sheet through a slit die and rapidly quenching the extruded or coextruded web upon a chilled casting drum so that the polymeric component(s) of the sheet are quenched below their solidification temperature.

[0045] The quenched sheet is then biaxially oriented by stretching in mutually perpendicular directions at a temperature above the glass transition temperature of the polymer(s). The sheet may be stretched in one direction and then in a second direction or may be simultaneously stretched in both directions. The preferred stretch ratio in any direction is at least 3:1. After the sheet has been stretched, it is heat set by heating to a temperature sufficient to crystallize the polymers while restraining to some degree the sheet against retraction in both directions of stretching.

[0046] The polymer sheet may be subjected to any number of coatings and treatments, after extrusion, coextrusion, orientation, etc. or between casting and full orientation, to improve its properties, such as printability, barrier properties, heat-sealability, spliceability, adhesion to other supports and/or imaging layers. Examples of such coatings can be acrylic coatings for printability, polyvinylidene halide for heat seal properties, etc. Examples of such treatments can be flame, plasma and corona discharge treatment, ultraviolet radiation treatment, ozone treatment and electron beam treatment to improve printability and adhesion. Further examples of treatments can be calendaring, embossing and patterning to obtain specific effects on the surface of the web. The polymer sheet can be further incorporated in any other suitable support by lamination, adhesion, cold or heat sealing, extrusion coating, or any other method known in the art.

[0047] A preferred application of the invention is in imaging elements, including those utilizing photographic, electrophotographic, electrostatographic, photothermographic, migration, electrothermographic, dielectric recording, thermal dye transfer, inkjet and other types of imaging. A more preferred application of the invention is in photographic imaging elements, including photographic papers and films. Most preferred application of the invention is in photographic image display products, particularly those comprising a reflective support, which in turn comprises any material such as, natural paper, synthetic paper, unvoided polymers, voided polymers including polymeric foam, microvoided

Conductive agents

Ionic conductive agents:

[0059] Combination of polyethylene ether glycol Carbowax 3350 supplied by Union Carbide and lithium nitrate in a dry weight ratio of 40:60.

Electronic conductive agents:

[0060] Acicular antimony doped tin oxide dispersion, eg. FS 10D supplied by Ishihara Techno Corp.

[0061] Zinc antimonate colloidal dispersion, eg. Celnax™ CX-Z300H supplied by Nissan Chemical Industries

Inorganic oxide sol

[0062] Alumina modified colloidal silica, eg. Ludox™ AM supplied by DuPont

SAMPLE PREPARATION

[0063] Layers are coated from aqueous solutions of various compositions on to a photographic paper support comprising a paper core laminated on both sides with biaxially oriented polyolefin based sheets. This photographic paper support is similar to Sample C (invention) of U.S. Pat. No. 6,232,056 but without the Fusible layer (L7) and Writable/conductive layer (L8). The surface on which the aforesaid aqueous solutions of various compositions is coated is a biaxially oriented polypropylene based terpolymer, similar to the matte surface of BICOR 70 MLT supplied by Exxon-Mobil Corporation (vide, for example, U.S. Pat.No.5,853,965 for specifics). The terpolymer surface is corona discharge treated, followed by hopper coating of the coating solutions, and subsequent drying by hot air at or below 180°F.

TEST METHODS

[0064] For resistivity tests, samples are preconditioned at 72°F under specific relative humidity (RH) for at least 24 hours prior to testing. Surface electrical resistivity (SER) of the coated antistatic layer is measured with a Keithly Model 616 digital electrometer using a two point DC probe by a method similar to that described in US Patent number 2,801,191.

[0065] For backmark retention tests on photographic paper, a printed image is applied onto the coated antistatic layer using a dot matrix printer. The paper is then subjected to a conventional developer for 30 seconds, washed with warm water for 5 seconds and rubbed for print retention evaluation. The following ratings are assigned for backmark retention (BMR), with a rating of 1-3 being indicative of acceptable performance:

1= Outstanding, very little difference between processed and unprocessed appearance.
2= Excellent, slight degradation of appearance
3=Acceptable, medium degradation of appearance
4= Unacceptable, serious degradation of appearance
5= Unacceptable, total degradation.

[0066] For spliceability, a splice is made between two strips of photographic paper, with the antistatic layer on one strip being in contact with the photographic emulsion on the other strip, as described in U.S. Pat. No. 6,171,769. Splicing is carried out using a splicing module used in commercial photofinishing equipment such as the Gretag CLAS 35 printer. The peel strength of the resultant splice is determined in an Instron machine, as a measure of spliceability.

[0067] Dust generation is assessed by means of a frictional wear test. A 1474 gram weight having three round rubber feet 0.25 inches in diameter (66 psi per foot) is placed on a black sheet of paper. The paper and weight are placed on top of the antistat coating and dragged over a distance of 10 inches back arid forth 5 times (total dragged distance of 50 inches). The dust generation is subjectively rated from visual inspection of the amount of material transferred to the black paper. The rating scale is as follows:

1 = no transfer
2 = barely visible transfer
3 = easily visible transfer, no distinct deposits
4 = easily visible transfer, distinct deposits
5 = large, elongated deposits of transfer

4-6 are also substantially higher than that of Comp. B. These results demonstrate that CPO without any additional conductive agent can provide an adequate antistatic layer, together with other desired properties (e.g., splice strength, BMR, dusting), for application in photographic paper whereas the polymeric binder of U.S. Pat. No. 6,171,769 lacks the necessary conductivity. Although Ex 4-6 perform reasonably well and better than the prior art (Comp. B), for superior performance the composition of the invention including additional conductive agent is preferred.

[0072] Samples Ex.7-9 were prepared similar to sample Comp.A, except that the polymeric binder Neocryl A5045 was blended with a CPO, namely CP349W, in 5/95, 10/90 and 20/80 weight ratio, respectively. The details these samples and the corresponding test results are listed in Tables 3A and 3B, respectively. For ease of comparison, the test results of sample Comp.A are also included in Table 3B.

Table 3A

Sample	$LiNO_3$ Dry wt.%	Carbowax Dry wt.%	Ludox Dry wt.%	CPO (CP349W) Dry wt.%	Neocryl A5045 Dry wt.%	CPO/ Neocryl Wt. ratio	Coverage g/ m^2
Ex.7	4.6	3.1	18.5	3.7	70.1	5/95	0.3
Ex.8	4.6	3.1	18.5	7.4	66.4	10/90	0.3
Ex.9	4.6	3.1	18.5	14.8	59.0	20/80	0.3

Table 3B

Sample	SER, log Ω/□ 60%RH	Splice strength g
Comp. A	9.3	226
Ex.7	9.0	801
Ex.8	8.8	1409
Ex.9	8.1	1980

[0073] It is very clear that the blending of even a small amount of CPO can greatly improve the splice strength and SER of an antistatic layer, such as one taught in U.S. Pat. No. 6,171,769. This demonstrates the superiority of the present invention over some of the prior art.

[0074] Samples Ex. 10-13 were prepared in accordance with the present invention, using various CPO and electronically conductive particles such as zinc antimonite or acicular tin oxide. The details about these samples and the corresponding test results are listed in Tables 4A and 4B, respectively.

Table 4A

Sample	Sample	Electronic conductor Dry wt.%		CPO Dry wt.%	Coverag e g/m^2
RC58276		Zinc antimonite CelnCx-CXZ300H	Acicular tinoxide FS-10D		
18	Ex.10	75		25 (CP310W)	0.3
19	Ex.11	75		25 (CP347W)	0.3
20	Ex.12	75		25 (CP349W)	0.3
10	Ex.13		25	75 (CP310W)	0.3

Table 4B

Sample	SER, log Ω/□			Splice strength g	BM R	Dusting
	20%RH	50%RH	80%RH			
Ex.10	8.7	8.7	8.8	1268	1-2	2
Ex.11	9.2	9.2	9.3	1124	1-2	2
Ex.12	9.2	9.2	9.4	1179	1-2	2

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